

oratory by melting electrolytic iron and nickel in a vacuum furnace and annealing in vacuum at 900°C and a similar alloy after hydrogen annealing to reduce the oxygen content from a few hundredths to a few thousandths of a per cent. The latter has received the name "Hipernik." Here the maximum permeabilities and hysteresis losses are in the ratio of 20 to 1, the former having recently reached a value of 167,000.

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HYDROGENIZED IRON

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SINGLE crystals of iron grown by a new method<sup>1</sup> with high temperature heat treatments in hydrogen were found to have better magnetic characteristics than single crystals grown at lower temperatures in hydrogen<sup>2,3</sup> or in vacuum.<sup>4</sup> Experiments showed that the improved

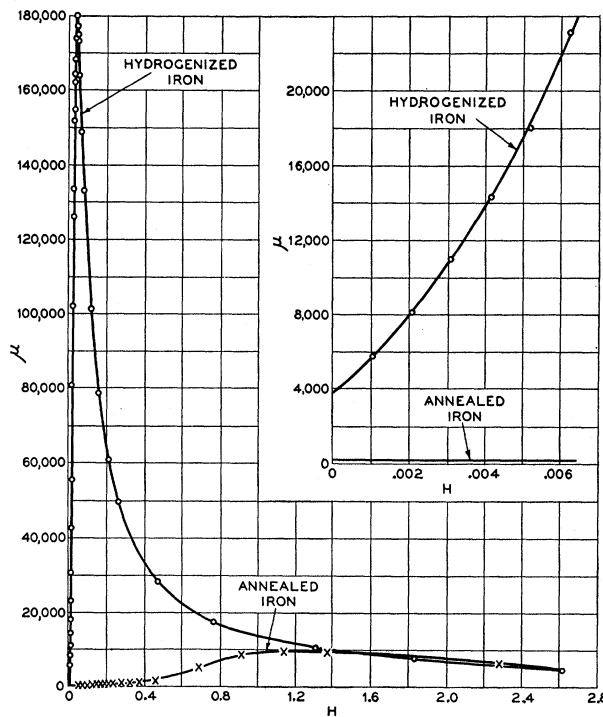


Fig. 1.

characteristics were not due to the large grain size but rather to the high temperature hydrogen treatment. It is now possible to produce iron having very high initial and maximum permeability regardless of grain size and orientation. The most recent results are shown in Figs. 1 and 2 for a ring of Armco iron 1-7/8" outside diameter, 1-9/16" inside diameter and 1/4" high, heat treated in moist hydrogen at 1475°C for 18 hrs., cooled to 880°C in one hr., then annealed at 880°C for 12 hrs., followed by slow cooling to room temperature. The initial and maximum per-

<sup>1</sup> L. W. McKeehan, Nature 119, 705-706 (1927).

<sup>2</sup> W. Gerlach, Zeits. f. Physik 38, 828-840 (1926).

<sup>3</sup> K. Honda and S. Kaya, Tohoku Imp. Univ. Sc. Rep., 15, 721-753 (1926).

<sup>4</sup> D. D. Foster, Phys. Rev. 33, 1071 (1929).

meabilities for this specimen are 4,000 and 180,000 respectively. The coercive force is 0.025 gauss and the hysteresis loss for  $B_m = 14,000$  is 190 ergs/cm<sup>3</sup>/cycle. These results can be readily reproduced.

Early experiments with wires of one mm diameter showed that heat treatments at 1500°C for 30 min. give high initial and maximum permeabilities when the rate of cooling through the

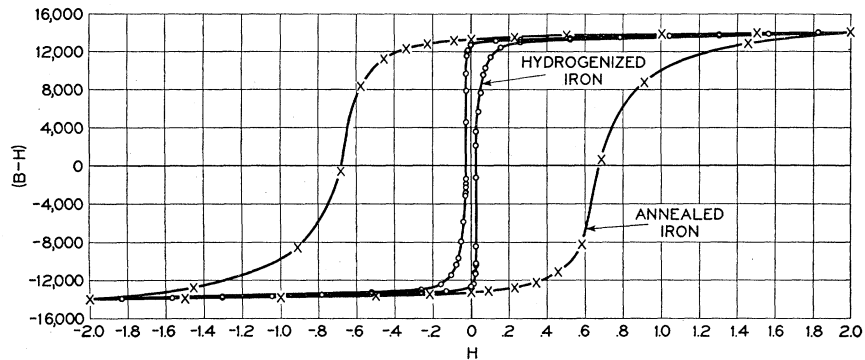


Fig. 2.

alpha-gamma transformation point is slow. By giving the hydrogen treatment in two steps: a high temperature treatment followed by rapid cooling, and a low temperature anneal below the alpha-gamma transformation point, a further improvement in magnetic characteristics is obtained. These results are shown in Fig. 3. It has been found that considerable overstrain may be applied between the high temperature treatment and the low temperature anneal without preventing good results.

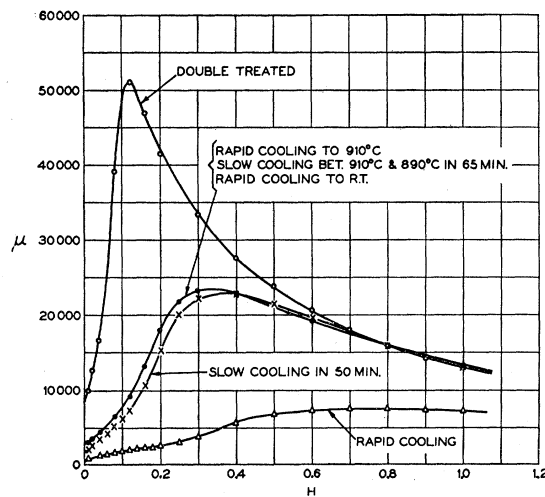


Fig. 3.

The magnetic characteristics of hydrogenized iron are dependent upon the cross-section of the metal, the maximum permeability rising rather rapidly with thickness as shown in Fig. 4. On the other hand, the required time of high temperature treatment increases with thickness of the metal, 5/32" thick specimens requiring as long as 18 hours.

Good results have been obtained by heat treatments at 1500°C in hydrogen at pressures as low as 45 mm and as high as 760 mm of mercury, but for pressures of 1 mm or 1520 mm the

results have been less satisfactory. There is reason to believe that there is an optimum pressure of hydrogen for every temperature of treatment.

High temperature heat treatments have been carried on in other atmospheres and in hydrogen containing various gaseous impurities. The most satisfactory results are obtained when the hydrogen is saturated with water vapor at room temperature.

Ordinary amounts of carbon, sulphur, phosphorus, oxygen and nitrogen, which are known to have harmful effects on the magnetic characteristics of iron, are reduced to very small quantities by prolonged hydrogen treatment. The large improvement obtained when water vapor is introduced in hydrogen is believed to be due to its increased effectiveness in decarburizing. E. D. Campbell<sup>5</sup> pointed out that moist hydrogen is most effective between 950° and

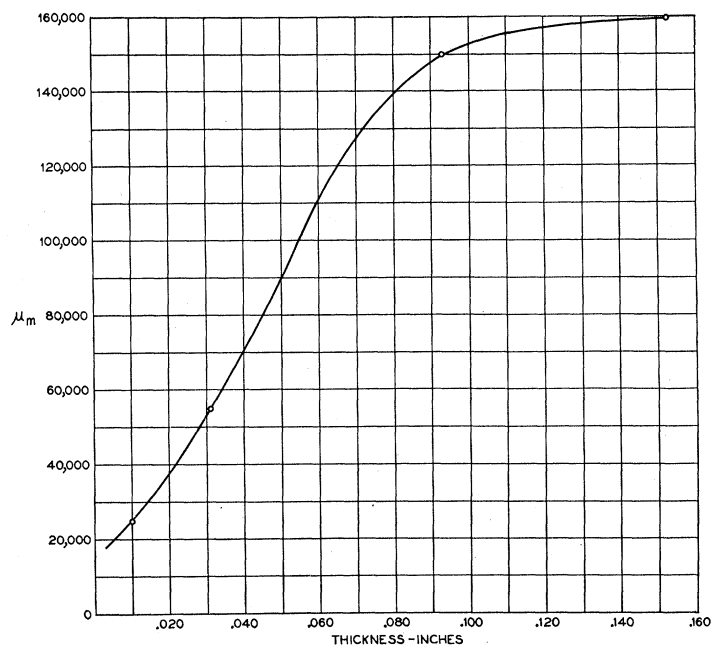


Fig. 4.

1000°C and since 1924 numerous experimenters, particularly those concerned with growing large single crystals of iron have treated their specimens at 950°C in moist hydrogen to remove the carbon which was supposed to inhibit grain growth. If decarburization plays an important part in these results, it appears as though temperatures higher than 950°C are much more effective. Ordinary impurities of silicon, manganese, copper and aluminum neither change in amount nor prevent good results. Vacuum treatments of hydrogenized iron cause a liberation of about  $\frac{1}{2}$  volume of gas, consisting of approximately 21 percent hydrogen, 33 percent CO<sub>2</sub>, 34 percent CO, 2 percent N<sub>2</sub> and 10 percent H<sub>2</sub>O. The quantity of carbon evolved as CO and CO<sub>2</sub> represents less than 0.002 percent.

Hydrogenized iron is nearly as soft as annealed copper. Its tensile strength is 14,000 lbs./in.<sup>2</sup> its yield point is 7,000 lbs./in.<sup>2</sup> and its Rockwell *B* hardness lies between +10 and -10. Another characteristic is that grain growth in hydrogenized iron is more pronounced than in ordinary iron, ranging from about 0.04" to 0.2". Some grains are more than 0.5" in length, occupying the full cross-section of the specimen. Toroidal specimens of 2" diameter have been obtained consisting of 5 or 6 single grains. There is evidence that the large grains grow at the high temperature and that under certain conditions retain their identity even after cooling

<sup>5</sup> E. D. Campbell, J. F. Ross and W. L. Fink, Jour. Iron and Steel Inst. 108, 179-185 (1923).

through two transformation points. In many respects, however, hydrogenized iron is no different from ordinary iron. For instance, the high temperature hydrogen treatment produces no appreciable change in resistivity, temperature coefficient of resistance, density and lattice parameter.

We do not know yet in what manner the high temperature hydrogen treatment produces the remarkable results described. It is well known that the magnetic characteristics of iron are improved by purification.<sup>6</sup> Chemical analyses show that the high temperature treatment does purify, and further evidences of this purification are large grain growth and mechanical softness. It is plausible therefore to suggest that purification plays a considerable part in these results. On the other hand the dependence of the magnetic characteristics of hydrogenized iron on the pressure of hydrogen, rate of cooling and time of anneal at 880°C are consistent with the view that absorbed hydrogen also plays an important part in these results.

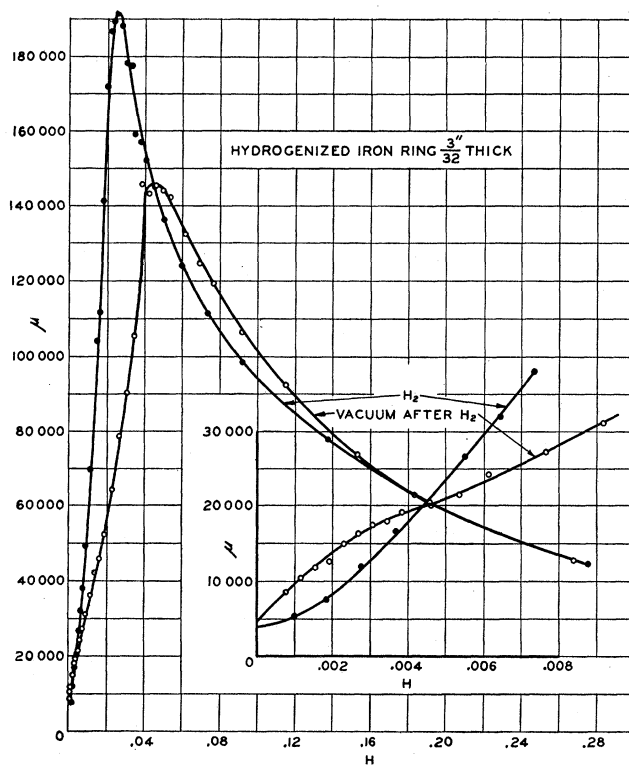


Fig. 5.

It was reported in a previous note<sup>7</sup> that further purification of hydrogenized iron by heat treatment in a high vacuum causes a diminution in permeability. This diminution was believed to be due to the pumping out of absorbed hydrogen. Since then, it has been found that the magnetic characteristics are dependent upon thickness, the permeability diminishing with decrease in thickness, and the previously reported diminution in permeability can now be largely accounted for by the decrease in thickness of the specimen produced by excessive evaporation during the vacuum treatment. In a more recent experiment, a specimen  $\frac{3}{32}$ " thick having a maximum permeability of 190,000 was heat treated in vacuum at 1200°C for 100 hours and at 880°C for 24 hours. The decrease in maximum permeability to 145,000 as shown in Fig. 5, can

<sup>6</sup> T. D. Yensen, Jour. Franklin Inst. 206, 503-510 (1928).

<sup>7</sup> P. P. Cioffi, Nature 126, 200-201 (1930).

also be largely accounted for by the decrease in thickness produced by evaporation. It now appears as though what previously seemed to be an effect ascribed to the pumping off of absorbed hydrogen may have been an effect due to decrease in thickness. It is just possible, however, that for this thickness of metal the 100 hours treatment in vacuum may have been insufficient to cause all of the absorbed hydrogen to diffuse out. The change in shape of the initial part of the permeability curve suggests inhomogeneity possibly due to a concentration gradient of absorbed hydrogen. In any case, a high vacuum treatment, supposedly producing further purification of hydrogenized iron has not been found to produce a further increase in permeability.

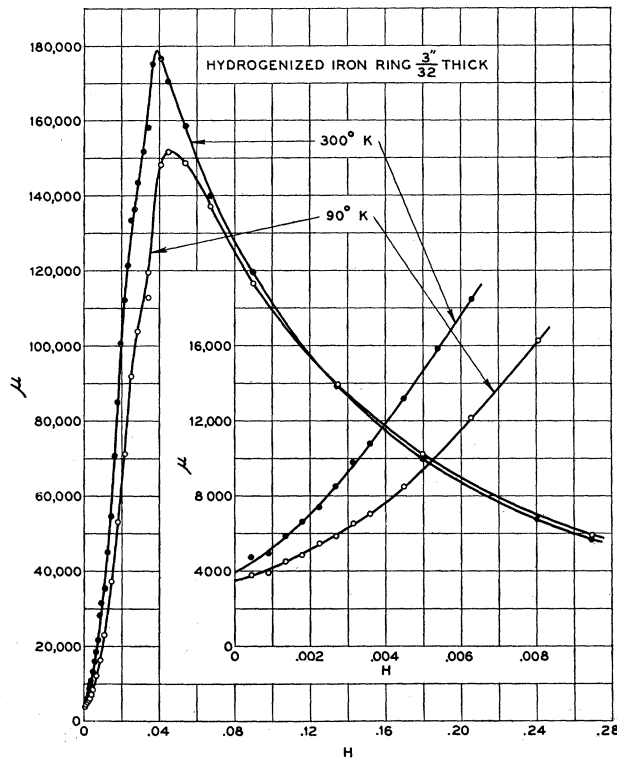


Fig. 6.

Grain size and thickness of specimen affect the magnetic characteristics as soon as the grains become so large or the specimen so thin that only a few grains occupy a cross-section perpendicular to the applied magnetic field. This has been checked in several ways. Under these conditions the flux distribution becomes non-uniform and the average permeability of the specimen drops.

The effect of lowering the temperature of hydrogenized iron to that of liquid air is shown in Fig. 6. The relatively small diminution in permeability accompanying a large decrease in temperature makes it appear unlikely that the characteristics of hydrogenized iron are due to thermal impacts between the absorbed hydrogen and iron atoms.<sup>8,9</sup>

October 15, 1931.

<sup>8</sup> L. W. McKeehan, Nature 126, 952-953 (1930).

<sup>9</sup> K. Honda, Zeits. f. Physik 67, 808-811 (1931).